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BENEFICIATION OF GRAPHITE FROM QUEBEC (PROJECT MP-IM-6601)

by

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SUMMARY

A study of the beneficiation of a graphite sample from Quebec was undertaken at the request of Mr. Gordon T. McMichael, Q.C., for Portland Industrial Minerals Ltd/Portland Graphite Syndicate.

Flotation with pine oil, screening, grinding and further flotation gave flake graphite products from 85% carbon at 85.5% recovery to 92% carbon at 92% recovery. Size distribution in the products was such that a crucible-grade flake graphite, as specified by the Joseph Dixon Crucible Company, could be obtained with minor adjustments.

Dry beneficiation of the sample did not give promising results.

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MICROMEDIA

INTRODUCTION

Late in 1965, Mr. Gordon T. McMichael, Q.C., of McMichael, Wentzell & Gautreau, Ottawa, acting for Portland Industrial Minerals Ltd/ Portland Graphite Syndicate, submitted a sample of graphite from Ste. Therese de Gatineau, Quebec, for examination. A preliminary study of the material was favourable. A full-scale investigation of its beneficiation was undertaken, to recover crucible grade (80-90% graphitic carbon with a specified flake distribution) and other grades of graphite. The work described in this report was done on an 800 lb sample, received March 14, 1966.

DESCRIPTION OF SAMPLE

The sample consisted mainly of lump material of up to 6 to 7 inches in size.

Mineralogical examination indicated that diopside was the principal gangue mineral, with smaller amounts of feldspar sphene and calcite being present. Some mica occurred as distinct fragments of mica schist. A fluorine bearing silicate, clinohumite, was detected.

Graphite content was approximately 7 to 8.5%.

The graphite did not appear "oily" - it "smeared" or "marked" very litte.

ANALYSIS

Acid solubles and ash* were determined chemically - graphite content was then calculated as the difference. During ash analysis, the material was hard to fire and required an extended period in the furnace to burn off residual graphite -800°C for 48 hours or longer in an oxidizing atmosphere.

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*Ash as referred to in the report is acid soluble free ash, run on the sample after the acid soluble constituents were removed.

The graphite concentrates were analyzed by X-ray diffraction, using the diffractometer and Guinier camera.

TEST WORK

The test work was divided into (1) dry and (2) wet beneficiation. These are described separately.

A. Dry Beneficiation

1. Air Tabling

In an attempt to eliminate some or all of the hard siliceous material and free the graphite, all the ore was reduced by a jaw crusher to about 1 inch maximum size. A representative sample was fractionated by screening (Table 1) and the fractions were examined visually.

TABLE 1

Screen Analysis
Jaw Crushing

Fraction	Wt. %
+ 1 in.	13.4
- 1 in. + 3/4 in.	18.6
-3/4 in. + 1/2 in.	14.8
-1/2 in. + 4 m	17.8
- 4 + 8 m	6.7
- 8 + 14 m	7.5
-14 + 20 m	5.1
-20 + 28 m	4.0
-28 + 48 m	7.1
-48 + 65 m	1.8
-65 +100 m	1.4
-100 m	1.8
Total	100.0

Because little material free of graphite was evident, the ore was further reduced in size. Two methods of comminution were tried viz, a) Impact alone - the plus 4 mesh material, which constituted about 65 per cent of the sample, was passed through a hammermill, b) Impact plus rolls - the ore after reduction to minus 4 mesh in a hammermill, as in "impact alone", was sized by screening and each coarse screen fraction was passed through rolls set to break the rock and free the graphite.

The products from the comminution in (a) and (b) were kept separate for additional treatment. This consisted of screening sized fractions from the broken ore products, and feeding these to a Kipp Kelly air table to separate the free graphite from the rock.

Where impact alone was used to break the ore, fine size fractions were fed separately to the air table. Results are shown in Table 2.

TABLE 2

Air Tabling

Test No. 1: Graphite Products from Impact Crushed Feed

Fraction Mesh	Feed lbs	Product				
		Recovery oz	Analysis %	Graphite oz	Distribution % Test	Distribution % Overall*
- 4 + 8**	28.0	3	9.27	-	-	-
- 8 + 14	18.4	14	78.64	11	19.4	9.9
-14 + 20	14.3	19	87.77	16.7	29.4	15.1
-20 + 28	13.8	28.5	75.43	21.4	37.8	19.3
-28 + 35	6.6	9.75	81.04	7.6	13.4	6.8
-35	18.9	-	-	-	-	-
Total	100.0	71.25	79.6	56.7	100.0	51.0

* Based on head analysis: 6.90% graphite

**Not included in total calculation

The screen fractions from impact plus rolls crushing were air-tabled separately. However, to recover, liberated graphite quickly, and also to increase recovery, the air table tails from the coarse fractions were further reduced by rolls crushing, screened, and the fractions added to those already obtained from primary comminution. Middlings from air tabling were also recirculated. The screen sizes used and the products obtained are shown in Table 3.

TABLE 3

Air Tabling
Test No. 2: Graphite Products from Impact plus Rolls Crushed Feed

Fraction Mesh	Feed lbs	Product				
		Recovery oz	Analysis %	Graphite oz	Distribution % Test	Distribution % Total*
- 8 + 14	13.1	14.25	80.71	11.5	18.3	10.5
-14 + 20	24.6	25.75	71.76	18.5	29.5	16.8
-20 + 28	12.3	18.5	84.46	15.6	24.9	14.2
-28 + 35	7.4	9.75	71.77	7.0	11.2	6.4
-35 + 48	17.5	14.25	70.80	10.1	16.1	9.1
- 48	25.1	-	-		-	-
Total	100.0	82.5	76.0	62.7	100.0	57.0

*Based on head analysis: 6.90% graphite

2. Electrostatic Separation

To remove mica that collected with the graphite during air tabling, electrostatic separation of the sized product was tried as follows: negative roll, positive corona, discharge varied from 1000 to 13,000 V.

There was little, if any, indication that mica could be separated this way.

B. Wet Beneficiation

1. Flotation

A series of bench scale flotation tests was run. Conditions were varied until an acceptable product was obtained. Refinements led to improved recovery and grade.

Feed for flotation was prepared by dry comminution to minus 14 mesh. In the first reduction stage, the plus 4 mesh fraction was screened out and reduced to minus 4 mesh in a hammermill. In the second reduction stage the plus 14 mesh fraction was screened out and reduced to minus 14 mesh by rolls crusher. A screen analysis of the minus 14 mesh flotation feed is given in Table 4.

TABLE 4

Screen Analysis
Flotation Feed

Fraction Mesh	Wt. %
+14	2.1
-14 + 28	41.2
-28 + 35	17.5
-35 + 48	12.6
-48 + 65	9.4
-65 +100	6.4
-100	10.8
Total	100.0

Pine oil and pine oil plus kerosene were tried as promoting reagents. The kerosene brought over more rock particles with small inclusions of graphite.

Using pine oil alone, and cleaning three times, graphite concentrates with over 80% carbon were obtained. These concentrates were further upgraded by passing them through the Jones Wet Magnetic Mineral Separator (1) at 25 amps. A typical result is given in Table 5.

TABLE 5

Flotation and Magnetic Separation

Reagents: Pine oil, 0.4 lb/t to rougher, 3x0.1 lb/t to cleaner						
Test No.	Fraction	Wt %	Acid Sol %	Ash %	Graphite	
					% (by diff)	Dist %
6	Rougher Tails	88.0	8.93	90.35	0.72	8.0
	1st Cl Tails	3.3	6.43	66.04	27.53	11.2
	2nd Cl Tails	1.3	1.96	43.69	54.35	8.7
	3rd Cl Tails	0.5	1.37	44.99	53.64	3.3
	Mags	0.1	5.42	38.40	56.18	0.7
	Midds	1.8	0.94	18.90	80.16	17.9
	Non mags	5.0	0.83	17.94	81.23	50.2
	Total (calc)	100.0	-	-	8.09	100.0

Graphite determinations were obtained on the screened products of three flotation tests as shown in Table 6. It is clear that the coarser fractions contain the most graphite.

TABLE 6

Screen Analysis
Flotation Concentrates

Test No.	Screen Fraction (mesh)	Acid Sol %	Ash %	Graphite %(by diff)
7	+48	0.58	6.91	92.51
	-48 + 65	0.61	10.30	89.09
	-65 +100	0.85	15.74	83.41
	-100	1.35	20.41	78.24
8	+35	0.27	2.47	97.26
	-35 + 48	0.22	8.24	91.54
	-48 + 65	0.93	23.80	75.27
	-65 + 100	1.50	32.14	66.36
	-100	1.87	28.87	69.26
9	+28	0.21	2.55	97.24
	-28 + 48	0.28	11.92	87.80
	-48 + 65	1.14	29.34	69.52
	-65 + 100	1.70	37.77	60.42
	-100	2.04	33.20	64.76

Following this lead, a test was performed in which as much graphite as possible was floated. Sodium silicate was used as a dispersing agent and pine oil as promoter. The float was gently wet screened on a 35 mesh screen and the plus 35 mesh material was set aside as product. The minus 35 mesh material was ground for 2 minutes in an Abbe mill at 50% solids. The ground product was refloated with pine oil and cleaned twice. Results are given in Table 7.

TABLE 7

Two Product Flotation

Reagents: Sodium silicate, 2 lb/t to conditioner Pine oil, 0.4 lb/t to rougher, 2 x 0.1 lb/t to cleaners						
Test No.	Fraction	Wt %	Acid Sol %	Ash %	Graphite %(by diff)	Graphite Dist %
20	Rougher Tails	88.4	9.02	89.66	1.32	14.7
	1st Cl Tails	3.1	8.41	89.92	1.67	0.7
	2nd Cl Tails	0.3	5.86	77.19	26.95	1.0
	+35M Rougher Float	5.8	0.60	21.32	78.08	57.1
	3rd Cl Float	2.4	0.70	11.34	87.96	26.5
	Total (calc)	100.0	-	-	7.95	100.0

A similar experiment was made, this time with pine oil only. The combined plus 35 mesh rougher and cleaned floats were separated into screen fractions for analysis (Table 8).

TABLE 8

Two Product Flotation and Product Fractionation

Reagents: Pine oil, 0.4 lb/t to rougher, 2 x 0.1 lb/t to cleaners						
Test No.	Fraction	Wt %	Acid Sol %	Ash %	Graphite %(by diff)	Graphite Dist %
24	Rougher Tails	89.8	8.64	90.14	1.22	13.5
	1st Cl Tails	1.9	5.53	91.71	2.76	0.6
	2nd Cl Tails	0.1	2.23	60.01	36.76	0.4
	Product (mesh)					
	+14	0.3	0.12	2.99	96.89	4.1
	-14 + 28	3.2	0.20	13.14	86.66	34.3
	-28 + 35	1.9	0.50	23.91	75.59	18.1
	-35 + 48	1.0	0.33	10.08	89.57	11.2
	-48 + 65	0.8	0.24	5.53	94.23	8.7
	-65 + 100	0.4	0.46	11.70	87.84	4.0
	-100	0.6	1.72	30.90	67.38	5.1
	Head (calc)	8.2	-	-	84.17	85.5
	Head (assay)		0.35	14.32	85.33	
	Total	100.0	-	-	8.19	100.0

The combined concentrate, corresponding to "Assay" in Table 8, was subjected to mineralogical examination*. No conclusive evidence of mica was found although interference from graphite in X-ray diffraction and Guinier camera techniques may have obscured small amounts of mica. Variable proportions of pyroxene, feldspar and scapolite were identified as contaminants.

The specifications for crucible grade graphite supplied by the Joseph Dixon Crucible Company include a requirement of between 80 and 90% graphitic carbon with a particle size distribution allowing some range. The carbon requirement is met in this test. Figure 1 indicates that the product is also close to the size distribution required.

*Mineralogical Report, July 21, 1966.

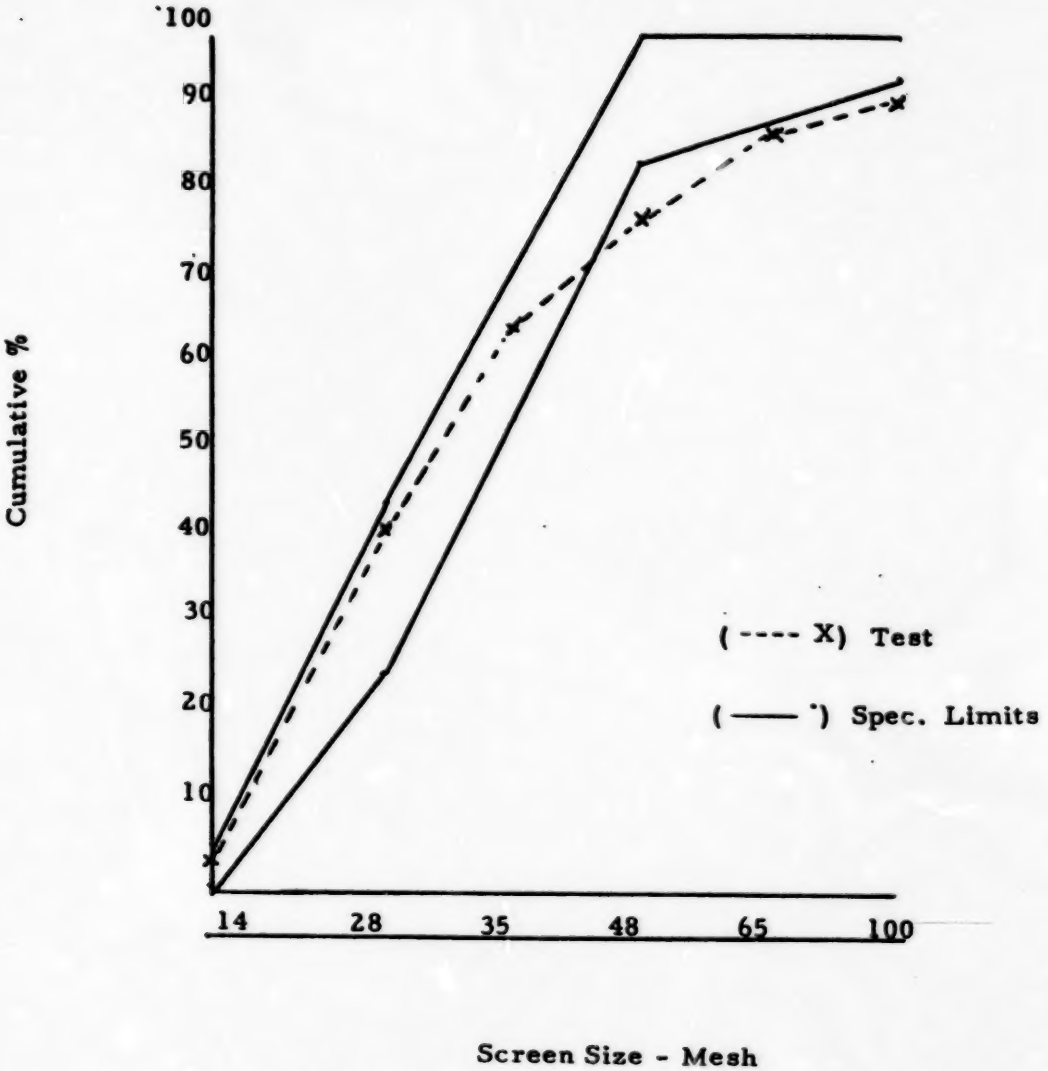

Figure 1. Comparison of Size Distribution with Specification Limits (Madagascar Crucible Flake).

The results shown in Table 8 indicate that the minus 28 plus 35 mesh fraction was low in grade compared to the other coarse screen fractions. A test was run floating as much of the graphite as possible with pine oil. This product was screened at 28 mesh rather than 35 mesh, the minus material was ground, refloated and cleaned. The two graphite products were combined, sampled and sized. Results are given in Table 9 and Figure 2.

TABLE 9

Two Product Flotation: Split at 28 Mesh

Reagents: Pine oil, 0.4 lb/t to rougher, 2 x 0.1 lb/t to cleaners						
Test No.	Fraction	Wt %	Acid Sol %	Ash %	Graphite	
					%(by diff)	Dist %
29	Rougher Tails	89.2	8.58	90.00	1.42	14.8
	1st Cl Tails	2.6	5.68	92.40	1.92	0.6
	2nd Cl Tails	0.2	6.45	65.77	27.78	0.6
	Product (mesh)					
	+ 14	0.4	0.11	3.24	96.65	4.5
	-14 + 28	3.3	0.21	12.07	87.72	34.4
	-28 + 35	1.5	0.20	10.31	89.49	15.9
	-35 + 48	1.1	0.18	4.26	94.56	11.8
	-48 + 65	0.7	0.17	3.29	96.54	8.0
	-65 + 100	0.4	0.27	3.54	96.19	4.1
	-100	0.6	1.51	20.85	77.64	5.3
	Head (calc)	8.0	-	-	89.88	84.0
	Head (assay)		0.32	9.83	89.85	
	Total	100.0	-	-	8.55	100.0

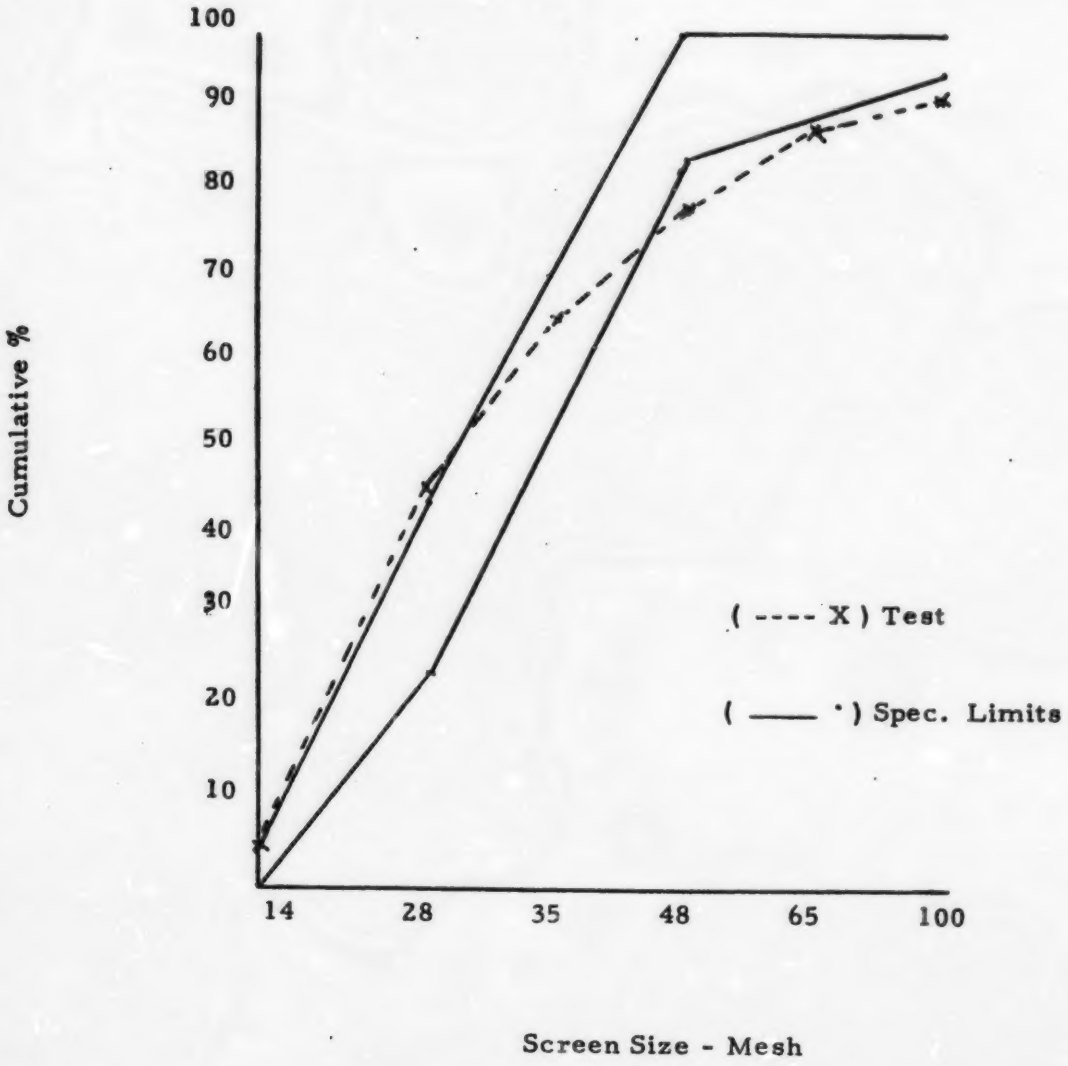

Figure 2. Comparison of Size Distribution with Specification Limits (Madagascar Crucible Flake).

To increase recovery, tests were run in which the rougher tails were (1) combined with the minus 28 mesh fraction before grinding and flotation and (2) ground separately and the freed graphite floated. These secondary treatments brought over a large amount of rock particles with small pieces of graphite attached.

Maximum recovery and grade were finally obtained with the following procedure: (1) Floating minus 14 mesh feed with 0.4 lb/ton pine oil added in one addition with no conditioning, (2) screening the plus 28 mesh product from the flotation concentrate, (3) regrinding rougher tails 15 minutes in an Abbe mili and refloating with 0.2 lb/ton pine oil added in one addition with no conditioning, (4) combining this second graphite concentrate with the minus 28 mesh material from the primary float, (5) regrinding for 5 minutes and refloating, and (6) re-cleaning once (0.1 lb/ton pine oil in each cleaner step). The recleaned product was combined with the plus 28 mesh product to form the final concentrate. Results are shown in Table 10 and Figure 3.

TABLE 10

Flotation with Regrinding and Recleaning

Reagents: Pine oil, 0.4 lb/t to Rougher (Primary); 0.2 lb/t to Rougher (Reground); 2 x 0.1 lb/t to Cleaners						
Test No.	Fraction	Wt %	Acid Sol %	Ash %	Graphite %(by diff)	Graphite Dist %
	Rougher Tails (Reground)	87.9	8.74	90.63	0.63	6.9
	Cleaner Tails	3.9	8.45	90.21	1.34	0.6
	Recleaner Tails	0.2	7.28	74.98	17.74	0.5
	Product (mesh)					
	+ 14	0.2	0.19	2.84	96.97	2.8
35	-14 + 28	2.7	0.24	10.60	89.16	30.7
	-28 + 35	1.5	0.20	7.61	92.19	16.8
	-35 + 48	1.3	0.20	3.25	96.55	15.8
	-48 + 65	0.8	0.18	2.86	96.96	10.0
	-65 + 100	0.7	0.29	2.98	96.73	8.1
	-100	0.8	1.68	24.15	74.17	7.8
	Heads (calc)	8.0	-	-	91.16	92.0
	Heads (assay)		0.27	7.71	92.02	
	Total	100.0	-	-	7.99	100.0

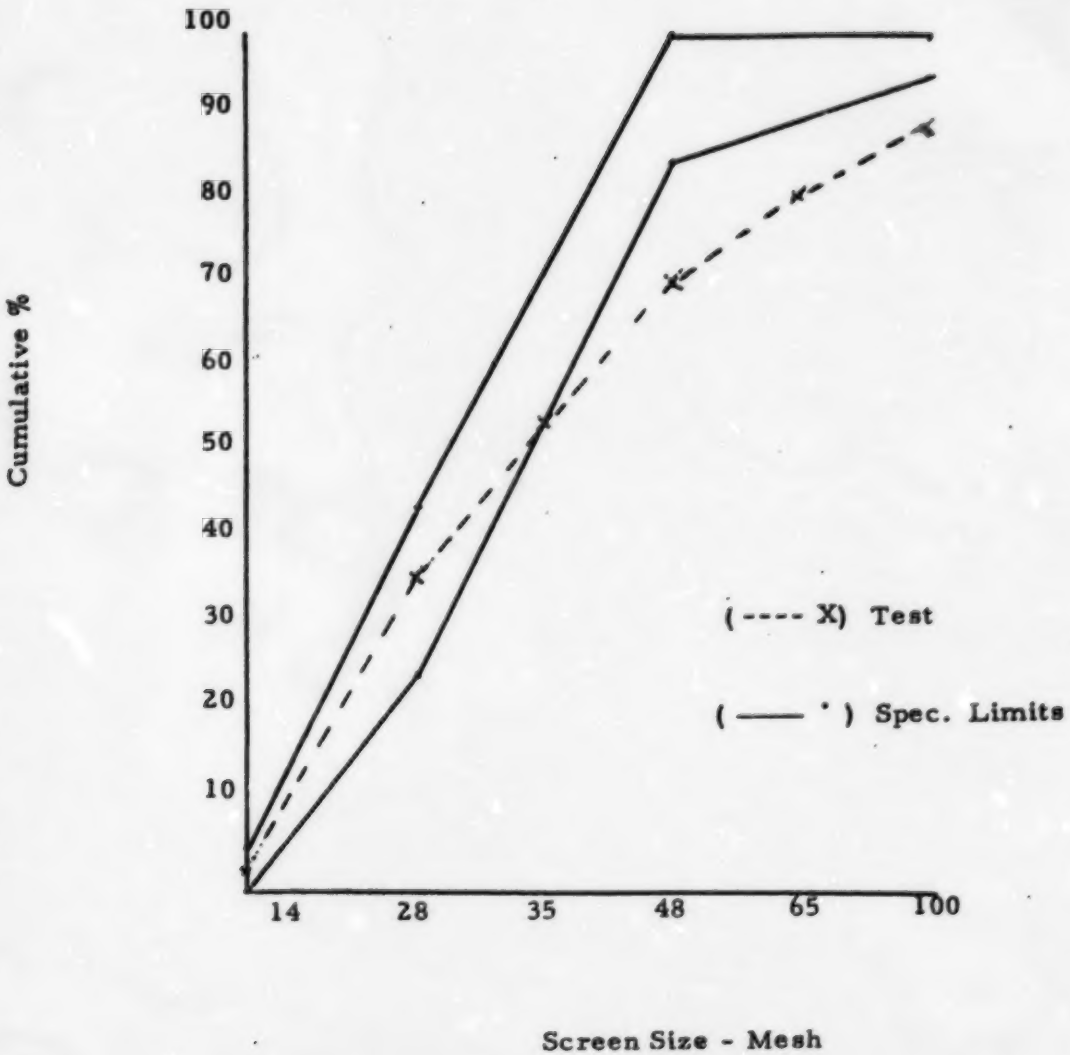

Figure 3. Comparison of Size Distribution with Specification Limits (Madagascar Crucible Flake).

REMARKS

The desired end product of this investigation was crucible-grade graphite. This should be free of mica and calcite. Specifications for size distribution and graphitic carbon content were supplied by the Joseph Dixon Crucible Company.

The graphite present in the sample appeared to be "tough" in the sense that it did not smear appreciably and lent itself to some grinding to free the flake from attached rock.

Dry beneficiation was not promising. With impact crushing, grade was just under 80% carbon-the specification's lower limit - and recovery was 51% (Table 2). When impact plus rolls were used as a means of comminution, grade was 76% carbon and recovery was 57% (Table 3). With an air table it is hard to handle the finer sizes. Further, any product would also include mica, if present, which would necessitate another type of treatment for its removal.

Electrostatic separation, which is one possible method of separating graphite from mica and waste rock, gave discouraging results.

Wet beneficiation using flotation with minus 14 mesh feed and pine oil as the only reagent gave a crucible-grade product, 85% carbon at 85.5% recovery (Table 8). Size distribution of the flakes was such that little adjustment would be required to meet specifications (Figure 1). The important step in the treatment was the removal, by screening, of the coarse flake in order that the finer sizes, which contained rock particles with graphite inclusions, could be ground to free the graphite for further recovery by flotation.

Other refinements in the screening-flotation technique gave a graphite concentrate analysing 89-90% carbon with an 84% recovery (Table 9, Figure 2).

To obtain maximum recovery, the rougher tails were reground and with additional treatment gave a 92% carbon product at 92% recovery (Table 10, Figure 3).

It was noted that the acid solubles were high in the finer fractions of products recovered in tests where the rougher tails were ground still further. This indicated the importance of grinding to free the graphite, but of taking care not to overgrind.

Although sodium silicate and kerosene were tried in flotation, there was no indication that either was beneficial.

Where the graphite product was passed through the wet magnetic separator (Table 5), the magnetics were high in acid soluble and ash content. This method could be used to upgrade a low-grade product or make a premium grade.

The size distributions of the flake products (Tables 8, 9 and 10) were fairly close to specifications (Figures 1, 2 and 3). If the minus 100 mesh material were removed, or if some of the coarser sizes were reduced somewhat by grinding, the required distribution should be obtained.

Since crucible-grade flake was made at a good recovery, work on a secondary product was not followed up. In practice the operating circuit would be run to meet demands and any material discarded by screening could be ground or otherwise treated.

CONCLUSIONS

1. Flake graphite products, analysing from 85% carbon at 85.5% recovery to 92% carbon at 92% recovery, can be obtained by flotation with pine oil, screening, grinding and further flotation.

2. Reagent consumption is 0.6 - 0.8 lb of pine oil per ton.

3. The size distribution on these products is such that they should, by screening and comminution if necessary, give a crucible-grade flake graphite as specified by the Joseph Dixon Crucible Company.

4. The final basis of suitable quality must be made by the crucible manufacturers.

5. Dry beneficiation of the graphite did not give promising results.

6. Wet magnetic separation, using equipment such as the Jones Mineral Separator, could be used to improve the grade of a flotation product.

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REFERENCE

1. R. A. Wyman, W.J.D. Stone and F. H. Hartman. "Illustrative Applications of the Jones Wet Magnetic Mineral Separator, Mines Branch Technical Bulletin TB 36, Department of Energy, Mines and Resources, Ottawa".

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